

DEVELOPMENT OF A MEMS PLATFORM FOR THE ASSESMENT OF REINFORCEMENT MECHANISMS OF CARBON NANOTUBES IN A POLYMER MATRIX

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Abstract

We present the design and fabrication of a MEMS platform that enables observation of fracture events, a propagating crack, in a model multi-walled carbon nanotube (MWCNT)-PMMA composite system. Preliminary results indicate that cracks can be driven large distances ($\sim 1 \mu\text{m}$) in a 200 nm thick PMMA thin film.

INTRODUCTION

The exceptional mechanical properties of carbon nanotubes (CNTs) make them excellent candidates for use as a reinforcing phase in polymer composite systems. However, in order to realize the true potential of these polymer composites a better understanding of the CNT-polymer interaction and load transfer characteristics needs to be developed. Previous attempts to characterize the reinforcement mechanisms of CNTs in polymer matrices have studied CNTs protruding from composite fracture surfaces created by local heating from a focused TEM electron beam. [1],[2] However, this method is limited because one can not image the fracture event while simultaneously driving the crack with the focused electron beam. Here, we present the design and fabrication of a MEMS platform that enables the simultaneous imaging and driving of a crack in a MWCNT-PMMA composite sample.

Device Design

The MEMS platform, Figure 1, consists of two major components: the MWCNT-PMMA composite sample and a micromachined heater fabricated on a silicon nitride membrane. The composite sample was assembled on top of the MEMS heater using the composite electric field guided assembly method (CEGA) developed by Chung *et al.*[3] This method allows for the fabrication of an aligned MWCNT array whose spacing is readily controlled by changing the ratio of a d_c to a_c electric field components. Once the MWCNT array is deposited, a thin PMMA layer is spun on the device, thereby forming a composite sample whose structure is well defined.

The MEMS platform also contains a micro-heater that is integrated into the substrate supporting the composite sample. The micro-heater locally heats the sample causing a steep temperature gradient, which supplies the necessary thermal stress to drive crack propagation. The micro-heater is fabricated out of a thin metal film and uses Joule heating. The heater has a serpentine layout, Figure 1, to maximize the surface area in contact with the polymer film. Finite element modeling (FEMLAB; Comsol, Inc.) of the micro-heater shows that a local temperature of 350°C (a temperature that is sufficient to degrade the structure of the polymer) can be achieved with an applied potential of 50 V. The low profile, planar design of the MEMS platform allows for observation of fracture events in either a transmission electron microscope (TEM) or an atomic force microscope (AFM).

Fabrication

The fabrication of the MEMS platform is a multi-step process that makes use of conventional surface micromachining techniques. The MEMS platform uses a 200 μm thick single crystal silicon (p-type with $\langle 100 \rangle$ orientation) as a substrate. First a 200 nm thick thermal silicon dioxide film, and then a 50 nm thick LPCVD low stress silicon nitride film, is grown on the silicon substrate. These films in combination serve as a masking layer during the KOH anisotropic etch process and as a membrane to support the PMMA during the fabrication of the composite sample. Optical lithography is used to define the specimen window on the backside of the silicon substrate, and the pattern is transferred into the wafer using reactive ion etching (RIE). A through wafer window is formed by anisotropic etching of the silicon substrate using 45% (by volume) KOH solution at 95°C for approximately 3 hours. The specimen window is necessary to allow for observation of the composite sample in the TEM and improves the efficiency of the micro-heater. Optical lithography with backside alignment is then used to define two sets of electrodes: one set is used by the micro-heater and the other is used in the deposition of the

MWCNT array. A thin chromium/gold (10/20nm) metal film is then deposited by electron-beam evaporation, and lift-off of the photoresist layer using acetone leaves the desired metal electrodes. Electron-beam lithography of a bi-layer resist (PMMA - Microchem Nano 495/Copolymer – Microchem MMA 8.5) is then used to form the serpentine shape of the micro-heater. A 30 nm thick chromium film is then deposited, and the heater is defined by lifting off the resist layer using acetone.

The composite sample is fabricated on top of the nanoscale heater by first depositing MWCNTs (previously grown by an arc-discharge method [4]) across opposing electrodes using the CEGA method. Once the MWCNTs are deposited, a PMMA polymer layer (Molecular Weight of 495k) is spin coated on the device, forming the composite sample. The final step is to pattern a pre-notch into the PMMA film using electron beam lithography. The pre-notch provides an initial crack where the thermal stress is maximized and provides a marker for the observation of crack propagation.

Conclusion

The observation of a propagating crack in this MWCNT-PMMA composite sample provides qualitative information about the reinforcement mechanisms of carbon nanotubes in polymer matrices. This approach may be useful in the future for developing a deeper understanding of the composite at the local (nanoscale) level. Preliminary results indicate that cracks can be driven large distances ($\sim 1\ \mu\text{m}$) in a 200 nm thick PMMA film using this device.

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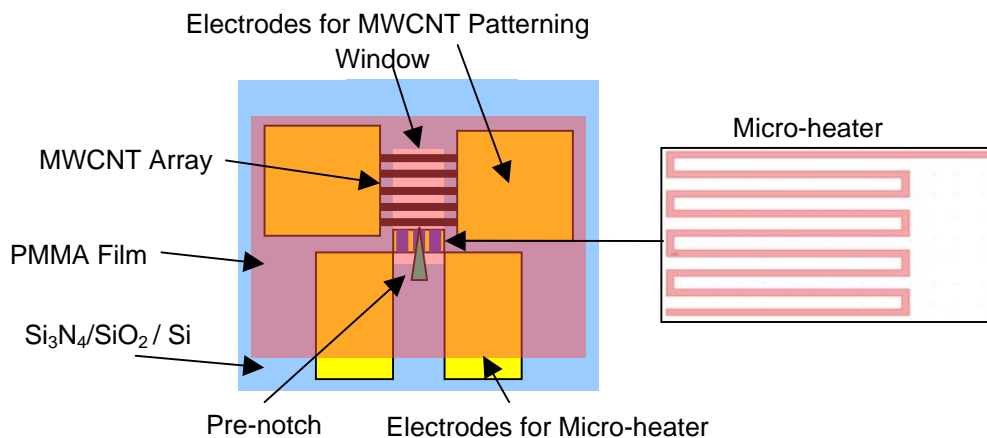


Figure 1. Schematic of the fabricated MEMS platform.

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